OPTIMIZATION OF RECYCLED FIBER IN LINERBOARD

Project 2697-53

Status Report

to

RAC Container and Board Subcommittee

March 20-21, 1979

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SUMMARY

This status report summarizes research in progress on ozonation treatments to increase the bonding potentials of recycled fiber. This portion of the program is supported by Institute funds. In general, the results indicate that

1. Commercially "cleaned" OCC pulps exhibit large increases in burst, tensile and other properties after ozonation in the same manner as the corrugated furnish used in current and past research in this project. This held true for stocks obtained before and after asphalt dispersion processes. Thus, any contaminants remaining in the stocks did not appear to affect ozonation.

2. When OCC was separated into liner and medium fractions and then ozonated, it appeared that both fractions responded about equally to the ozone treatment. This may be helpful in the event it is desirable to fractionate and treat only one fraction for economic reasons. Further work is in progress to evaluate blends of the untreated and ozonated liner and medium fractions.

3. Exploratory trials suggested that ozonation will proceed quite rapidly in an excess of ozone. Ways to utilize this approach are being considered.

4. In general, the optimum consistency for ozonation appears to be about 40-50%. However, at low O_3 consumption rates, operation in the 30-40% range may be feasible.

In addition to the above, research in progress on chemical treatments and additives to increase bonding is also discussed. This portion of the work is supported by FKBG funds. Among the chemical treatments being evaluated are caustic soda, sodium carbonate, hydrogen peroxide and oxygen.

In addition, evaluation trials will be carried out using "green" and "white" liquors at the usual mill concentration levels.

INTRODUCTION

The first part of this research program is concerned with the addition of composite old corrugated to linerboard. In general, the overall objective of the study is to develop ways of using increasing amounts of recycled fiber in linerboard without sacrificing board quality and productivity.

Both the Institute and Fourdrinier Kraft Board Group (FKBG) of the American Paper Institute are funding the program. The specific portions of the research to be supported by the Institute and the FKBG are outlined in the current editions of the research plan. This status report briefly summarizes and identifies the research in progress since the October 1978 meeting on both the Institute and FKBG portions of the program. Future research plans are also outlined.

The current and future research outlined herein has been reviewed in detail with the FKBG Reclaimed Fiber Utilization Committee. In general, the planned research to be carried out in calendar 1979 using FKBG funds received the committee's approval. The committee also strongly favored the planned research on ozonation of recycled fibers which is supported by Institute funds.

OZONATION OF RECYCLED FIBER
(IPC Funding)

The initial studies summarized in Progress Report One (Project 2697-53, September 24, 1978) entitled Effect of Ozonation on Recycled Fiber Properties indicated ozonation will significantly improve the physical properties of OCC. This improvement occurs without significant loss of freeness, fiber length, or apparent attendant pollution problems associated with alternative treatment or processing methods. Therefore, further studies are in progress to define and optimize the variables associated with the ozonation process. In addition, work is being carried out on commercial OCC to determine if the contaminants remaining after cleaning will affect ozonation. Accordingly, the following series of studies were initiated.

<u>Ozonation of Commercial OCC</u>

The following samples of commercially cleaned OCC were obtained:

1. <u>Menasha Corporation</u>: Before and after asphalt dispersion (A/D) process
2. <u>Chesapeake Corporation</u>: 75% OCC, 25% kraft corrugated cuttings
3. <u>Union Camp Corporation</u>
4. <u>Inland Container Corporation</u>: Before and after A/D process

The samples from the Menasha Corporation, Chesapeake Corporation and Union Camp Corporation have been evaluated and the results are briefly summarized herein. The evaluation of the samples from the Inland Container Corporation is in progress. Upon receipt, each pulp was centrifuged to remove excess water, fluffed, ozonated, formed into handsheets and the physical properties were determined in accordance with procedures established for the IPC model OCC as described in Progress Report One, Project 2697-53. Single trials at ozonation levels of approximately 2.3% and 4.5% ozone consumed based upon the o.d. weight of the fiber were performed.

These O_3 consumptions correspond to treatment times of 15 and 30 minutes. In previous trials on the Institute "model" OCC, treatment times ranging up to 90 minutes were employed. For these comparisons, it was believed that the shorter treatments and lower O_3 consumptions would be of more practical interest.

Table I tabulates the physical test data on the Menasha Corporation pulps. Figure 1 shows that the ozone consumptions obtained on the commercial OCC from Menasha were about the same as obtained on the Institute "model" OCC at equal treatment times. In Figure 1, the O_3 consumptions on the "before" A/D stock are plotted; however, similar results were obtained on the "after" A/D stock. The commercial OCC stocks also exhibited small decreases in freeness with increasing ozonation in the same manner as the Institute furnish used in previous work. Thus the small amounts of contaminants remaining in the commercial OCC did not change the O_3 consumption rates and freeness trends.

Figure 2 indicates that the tensile and bursting strengths obtained on the ozonated commercial OCC stocks from Menasha Corporation exhibit strength increases which are similar to those obtained on the IPC "model" OCC. The pulp obtained after the asphalt dispersion process exhibits lower burst and tensile strengths than the stock obtained after the A/D process as expected from the literature. However, the A/D process does not appear to affect the rate of improvement in burst and tensile with increasing ozonation. The

TABLE I

PROPERTIES OF HANDSHEETS PREPARED FROM COMMERCIAL OCC
OBTAINED FROM MENASHA CORPORATION

	Before AD Process			After AD Process			FKBG Composite OCC[a]		
Ozonation time, min	0	15	30	0	15	30	0	15	60
Ozone Applied, % of o.d. fiber	--	2.52	4.96	--	2.47	50.2	--	2.35	9.41
Ozone Consumed, % of o.d. fiber	--	2.44	4.66	--	2.29	4.75	--	2.31	8.53
C.S. freeness, cc	550	535	520	590	570	550	633	617	567
% Change	--	-2.7	-5.5	--	-3.4	-6.8	--	-2.5	-10.4
Basis weight, lb/M ft^2	13.8	13.3	13.5	13.2	13.4	13.4	13.2	13.2	13.5
Caliper, points	5.7	5.2	5.2	5.4	5.2	5.1	6.1	5.6	5.3
Apparent density	2.42	2.55	2.57	2.45	2.56	2.63	2.16	2.34	2.54
% Change	--	+5.4	+6.2	--	+4.9	+7.3	--	+8.3	+17.6
Bursting strength, psig	20.5	24.2	29.2	18.2	22.3	24.8	17.2	23.2	33.1
Factor	1.49	1.82	2.17	1.38	1.66	1.86	1.30	1.76	2.45
% Change	--	+22.2	+45.6	--	+20.3	+34.8	--	+35.4	+38.5
Mod. ring compression, lb/inch	4.0	4.1	4.1	3.8	4.0	3.9	3.8	4.0	4.8
Factor	0.290	0.312	0.305	0.285	0.302	0.292	0.284	0.307	0.353
% Change	--	+7.6	+5.2	--	+6.0	+2.5	--	+8.1	+24.3
Tear, grams	93.1	85.2	79.2	109.2	99.2	90.0	87.6	85.1	71.9
Factor	6.75	6.41	5.89	8.30	7.39	6.73	6.64	6.45	5.31
% Change	--	-5.0	-12.7	--	-11.0	-18.9	--	-2.9	-20.0
Tensile, lb/inch	13.1	15.6	16.6	11.3	13.5	15.1	12.4	15.6	20.0
Factor	0.95	1.17	1.24	0.86	1.01	1.13	0.94	1.19	1.48
% Change	--	+23.2	+30.5	--	+17.4	+31.4	--	+26.6	+57.4
Stretch, %	2.34	2.60	2.50	2.55	2.90	2.99	1.86	2.18	2.43
% Change	--	+11.1	+6.8	--	+13.7	+17.3	--	+17.2	+30.6
Et, lb/inch	1754	1926	2083	1512	1740	1918	1640	1946	2326
Factor	127.4	144.8	154.9	114.9	129.7	143.4	124.2	147.8	172.0
% Change	--	+13.7	+21.6	--	+12.9	+24.8	--	+19.0	+38.5
TEA, ft-lb/ft^2	2.7	3.6	3.6	2.6	3.5	4.1	2.0	2.9	4.1
% Change	--	+33.3	+33.3	--	+34.6	+57.7	--	+45.0	+105.0

[a] Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

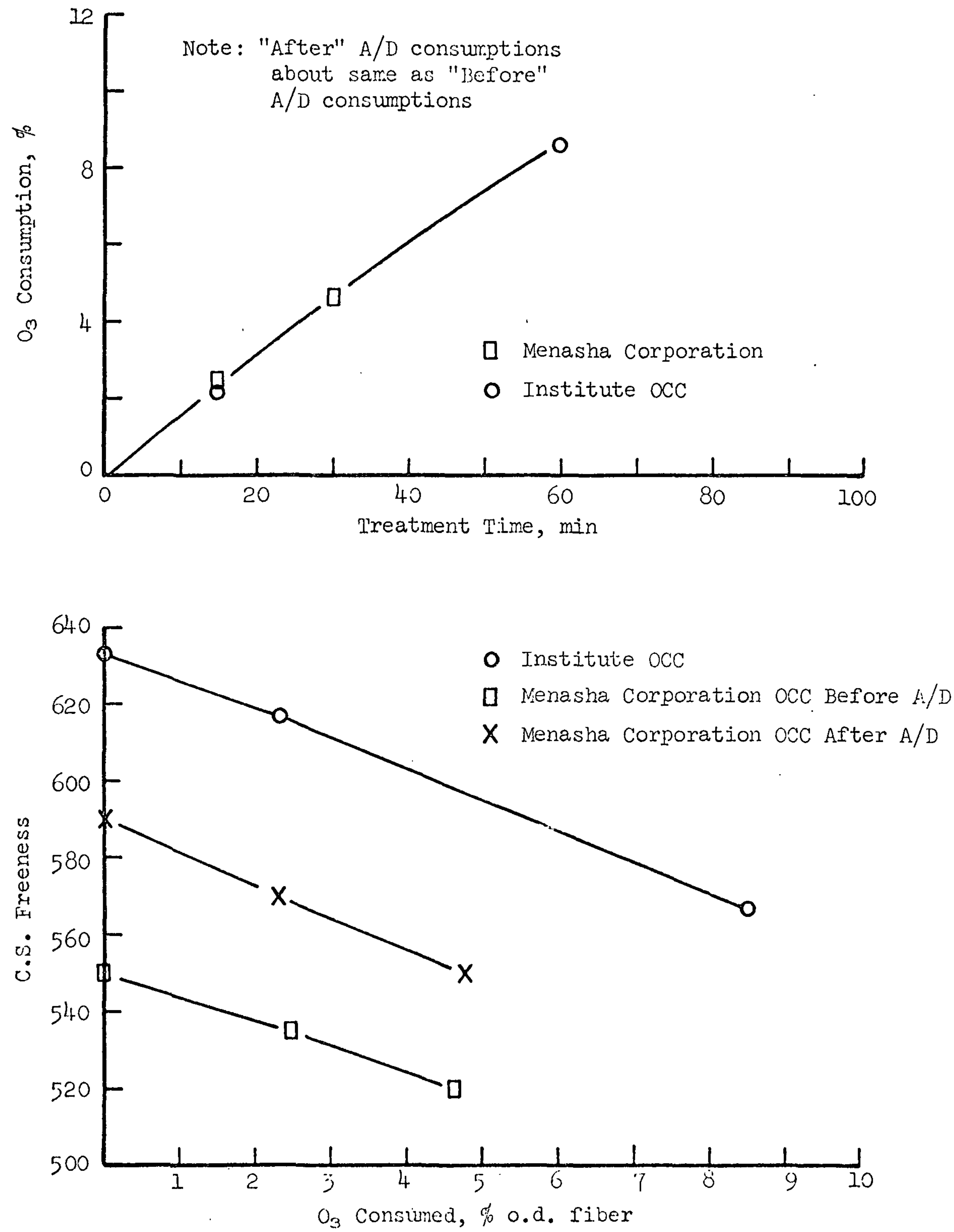

Figure 1. Comparison of Ozone Consumption and Freeness on Commercial OCC from the Menasha Corporation and Institute "Model" OCC

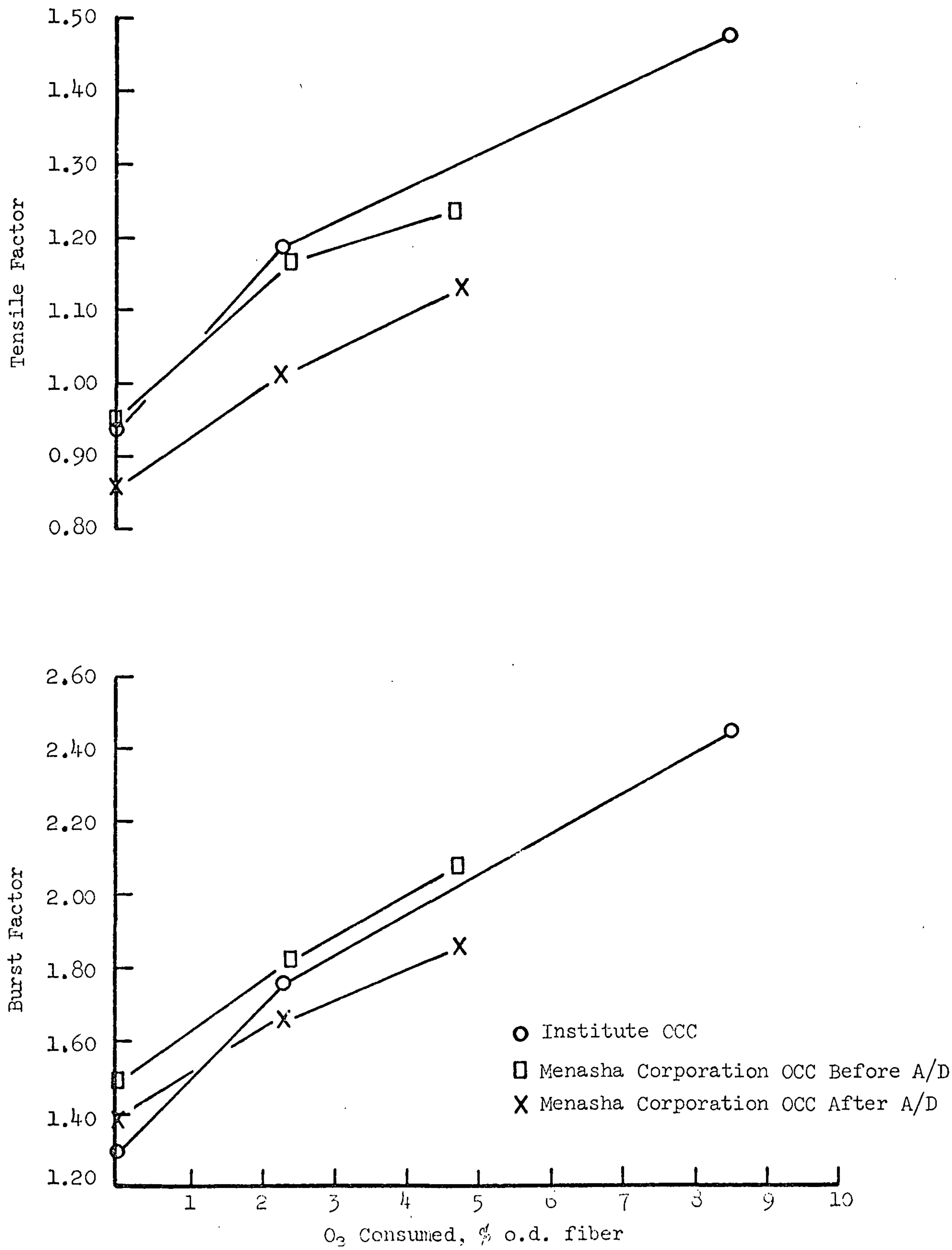

Figure 2. Comparison of Tensile and Burst Factors on Commercial OCC From the Menasha Corporation and Institute "Model" OCC

"before" A/D commercial OCC exhibited about the same strength levels as the IPC "model" stock.

Figure 3 shows that the rates of change of the tearing strengths of the commercially cleaned and ozonated OCC from Menasha Corporation were about the same as the ozonated IPC "model" OCC. The tearing strengths of the "before" A/D OCC were lower than the "after" A/D OCC and at the same level as the IPC "model" OCC at all ozonation levels.

The modified ring compression results in Figure 3 show that ozonation increased the strength of the commercial OCC stocks from the Menasha Corporation at the 2+% O_3 level in about the same amount as the IPC OCC. At the 4.5+% O_3 level, the ring strengths of the commercial OCC did not increase in the same way as the IPC stocks; however, compression tests on "thin" sheets are difficult to carry out and tend to be erratic. Review of the tabular data indicated that other physical properties follow the same trends with increasing ozonation as the Institute "model" OCC.

The Menasha Corporation pulp after application of the asphalt dispersion process exhibits an apparent real loss of strength when compared to the pulp before asphalt dispersion. However, application of the asphalt dispersion process does not appear to affect the rate of strength improvement upon ozonation. Initial properties of the Menasha pulp before asphalt dispersion more closely simulate the strength properties of the IPC model OCC.

Table II summarizes the results obtained on the commercially cleaned OCC stocks obtained from the Chesapeake Corporation and Union Camp Corporation. The ozone consumption levels obtained on these stocks were about the same as obtained on the Menasha and Institute samples (see Table I). Figure 4 shows

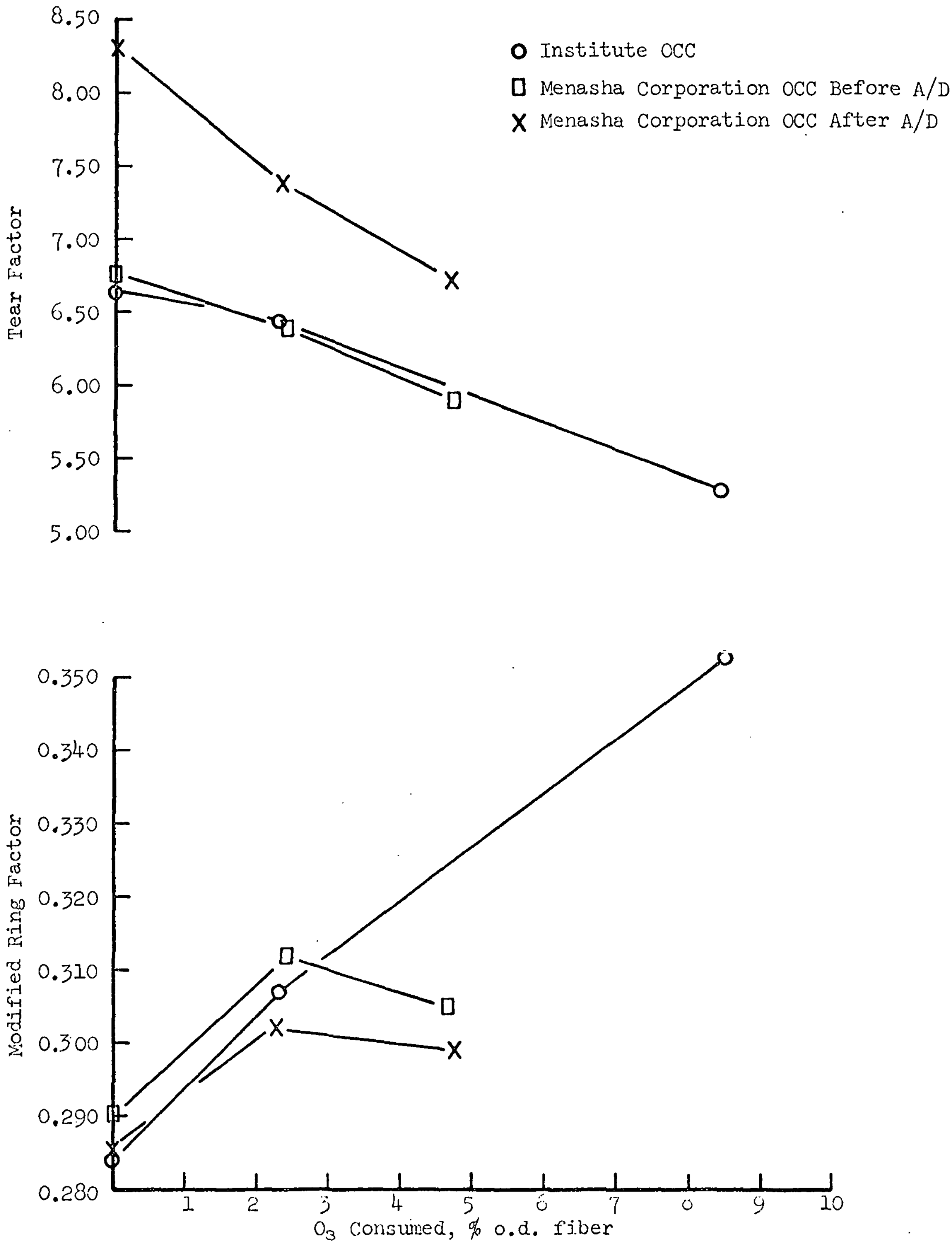

Figure 3. Comparison of Tear and Modified Ring Factors on Commercial OCC from the Menasha Corporation and Institute "Model" OCC

TABLE II

PROPERTIES OF HANDSHEETS PREPARED FROM COMMERCIAL OCC
OBTAINED FROM THE CHESAPEAKE CORPORATION AND UNION CAMP CORPORATION

	Chesapeake Pulp			Union Camp Pulp		
Ozonation time, min	0	15	30	0	15	30
Ozone applied, % o.d. fiber	---	2.54	5.04	---	2.58	5.16
Ozone consumed, % o.d. fiber	---	2.46	4.68	---	2.48	4.77
Reaction efficiency, %	---	96.5	92.8	---	96.0	92.4
C.S. freeness, cc	595	560	575	610	560	550
% Change	---	-5.9	-3.4	---	-8.2	-9.8
Basis weight, lb/M ft^2	13.5	13.7	13.4	13.5	13.9	13.5
Caliper, points	5.3	5.2	5.0	5.5	5.3	5.3
Apparent density	2.57	2.64	2.69	2.46	2.63	2.56
% Change	---	+2.7	+4.7	---	+6.9	+4.1
Bursting strength, psig	19.0	27.4	27.4	16.0	25.0	27.3
Factor	1.40	2.00	2.05	1.19	1.81	2.02
% Change	---	+42.9	+46.2	---	+52.1	+69.7
Mod. Ring Compression, lb/inch	3.8	4.3	4.3	3.6	4.1	3.9
Factor	0.277	0.317	0.320	0.267	0.299	0.292
% Change	---	+14.4	+15.5	---	+12.0	+9.4
Tear, grams	91.6	88.4	76.0	98.0	88.4	82.8
Factor	6.76	6.45	5.66	7.26	6.38	6.13
% Change	---	-4.6	-16.3	---	-12.1	-15.6
Tensile, lb/inch	13.3	16.3	16.1	12.3	15.2	16.6
Factor	0.98	1.19	1.20	0.91	1.10	1.23
% Change	---	+21.4	+22.4	---	+20.9	+35.2
Stretch, %	2.09	2.63	2.46	1.95	2.32	2.50
% Change	---	+25.8	+17.7	---	+19.0	+28.2
Et, lb/inch	1763	2023	2039	1690	1958	2058
Factor	130.2	147.6	151.9	125.2	141.3	152.4
% Change	---	+13.4	+16.7	---	+12.9	+21.7
TEA, ft-lb/ft^2	2.4	3.7	3.4	2.1	3.1	3.6
% Change	---	+54.2	+41.7	---	+47.6	+71.4

that the burst and tensile factors increased with increasing degree of ozonation in the same way as the OCC used in the Institute studies and the Menasha Corporation stocks.

Figure 5 shows that the rates of change of the tearing strengths of the ozonated stocks from Chesapeake Corporation and Union Camp Corporation were about the same as the Institute OCC. The modified ring compression trends in the commercially cleaned OCC stocks were also similar to those obtained in the Institute material although somewhat erratic at the 4+% O_3 level (Figure 5).

Briefly summarizing, these results indicate that commercially cleaned OCC pulps exhibit large increases in burst, tensile and other properties after ozonation in the same manner as the corrugated furnish used in the current and past research in this project. This held true for samples taken both before and after A/D processing. It also may be noted that fiber classifications of the commercial and Institute samples were quite similar.

Visual examinations of the fibers and handsheets (Menasha Corporation pulps) utilizing the SEM (scanning electron microscope) and optical microscopes were performed as a part of the overall study and generally compared favorably with the IPC model OCC. Several limited differences were noted. The Menasha pulp appeared to present a more "coated" surface upon SEM viewing. Pigment particles also were observed on the fiber surfaces. Limited evidence suggests that they are clay and titanium pigments. As a result, total ash was determined on the samples as well as the IPC model OCC. Results indicate that commercial OCC contains about 2.5% ash as compared with 1.3% for the model OCC. Total solubles and total organic carbon in the solubles are being considered as possible future tests in an effort to provide further characterization of

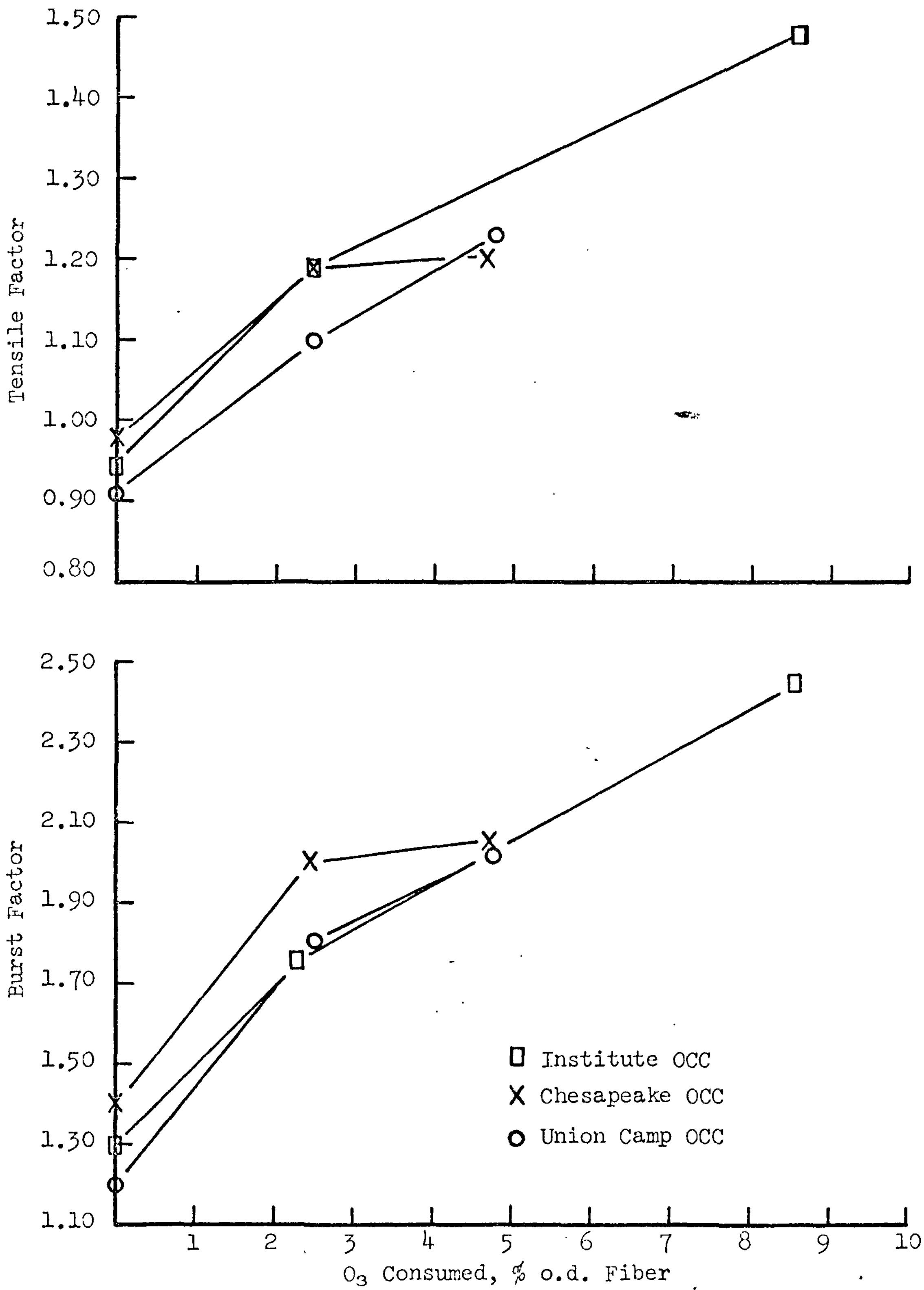

Figure 4. Burst and Tensile Strength on Ozonated Commercial OCC

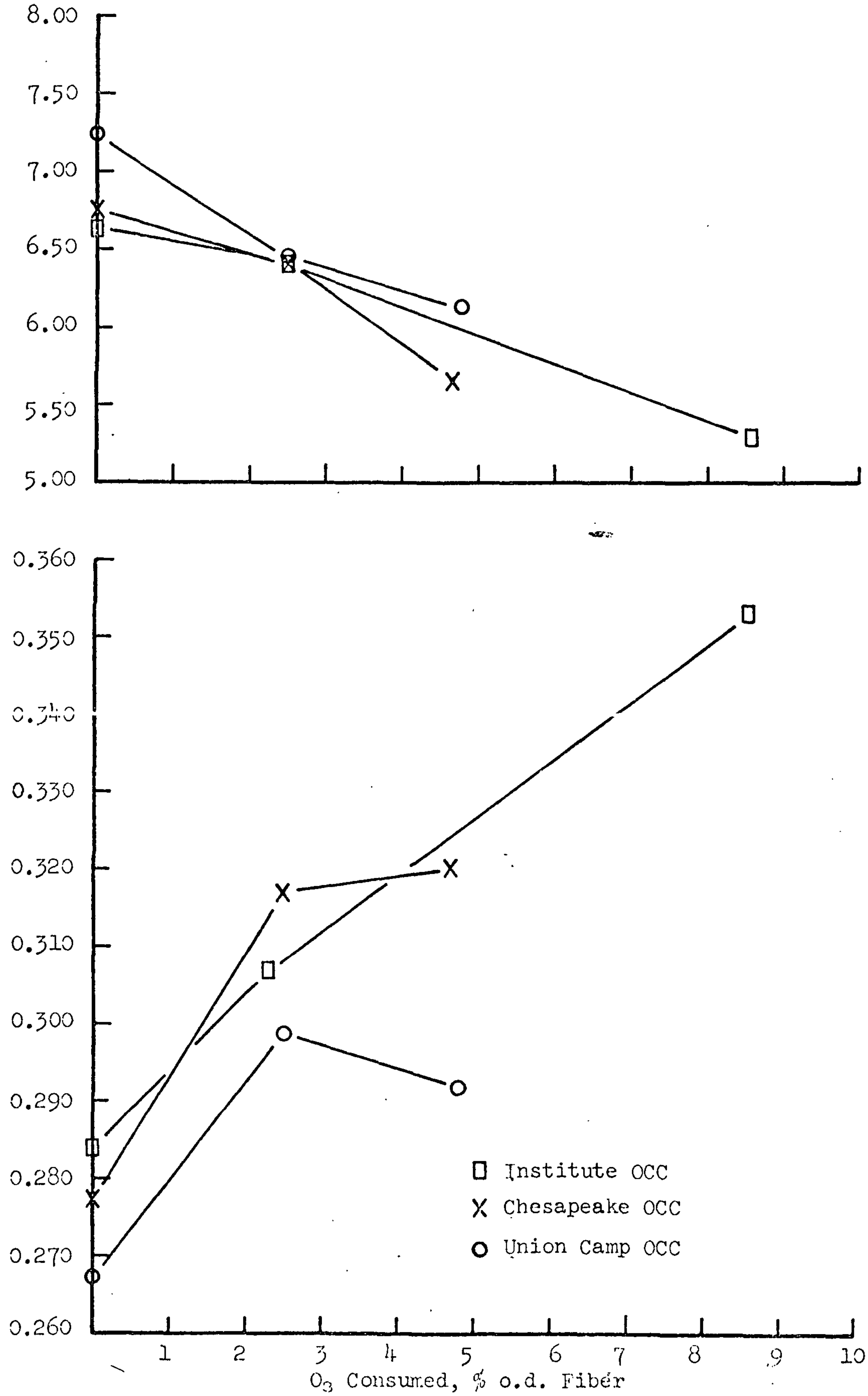

Figure 5. Tear and Ring Compression Strengths on Ozonated Commercial OCC

contaminants in commercial OCC. It is expected that significant solubles will be found in the commercial pulps based upon the observations of the water removed during centrifuging.

Fiber analysis indicates a broader array of pulps and species were found in the commercial material, including small quantities of bleached and unbleached softwood and hardwood sulfites and krafts and monocotyledon (probably straw). The major components of the furnish were softwood kraft and hardwood NSSC similar to the IPC model OCC. Ratios of the softwood and hardwood were similar to the IPC model OCC.

Ozonation of Liner and Medium Fractions

In previous studies the OCC composite was ozonated; however, it was hypothesized that ozonation of the long and short fiber fractions separately might be more effective in terms of overall strength improvements at reduced cost. For example, treatment of the medium fraction only could reduce ozonation costs by about two-thirds. Different pulp furnishes might also respond differently to ozonation. Previous C-stain fiber studies suggested such possibilities could occur. Initial studies with these potentials in mind were therefore undertaken.

The liner and medium fractions of the IPC model OCC were separated by soaking, disintegrated, fluffed, and ozonated in accordance with procedures previously established for the OCC composite. This separation yielded a liner fraction which is essentially 100% softwood kraft and a medium fraction of approximately 80% hardwood NSSC and 20% softwood kraft. Single trials at ozonation levels of approximately 2.3% and 4.5% ozone consumed based upon the pulp were performed. The fractions were formed into handsheets and the usual physical tests were performed.

Physical test results are shown in Table III. Graphs of freeness, tensile, burst, tear and modified ring vs. O_3 consumption are shown in Figures 6-8. Comparable results for the composite are also shown. The trends in percentage strength improvement for the liner and medium are very similar even though initial strength values for the liner and medium are quite divergent. This suggests that both furnishes respond about equally well to ozone treatment. However, it may still be desirable to fractionate and treat only one fraction for economic reasons and/or to avoid further fiber shortening and reduction of freeness. This would be particularly true for the hardwood furnish. Properties of the OCC composite generally are closer to the properties of the liner fraction as might be expected since approximately 70% of the composite is softwood kraft.

C-stain and SEM studies were undertaken. C-stain color shift trends with increasing ozonation were similar to those reported for the various softwood and hardwood components of the composite OCC, although responses to color shift to the blue appeared to occur at a slightly faster rate than for the comparable composite.

SEM studies exhibited some differences. The trend of "erosion" and fibrillation on the liner (softwood) fraction could be observed. However, these same trends were not readily observed on the medium (hardwood) fraction. Previous C-stain studies on the composite have shown that softwood earlywood appears to respond to ozonation before the hardwood fibers and may serve to "explain" the differences in fiber surface responses. However, the fact that

TABLE III

COMPARISON OF PROPERTIES OF HANDSHEETS PREPARED FROM OZONATED LINER AND MEDIUM FRACTIONS WITH COMPOSITE OCC

	Liner			Medium			Composite OCC[a]		
Ozonation time, min	0	15	30	0	15	30	0	15	60
Ozone applied, % of o.d. fiber	--	2.43	4.79	--	2.46	4.77	--	2.35	9.41
Ozone consumed, % of o.d. fiber	--	2.35	4.48	--	2.34	4.41	--	2.31	8.53
Reaction efficiency, %	--	96.7	93.5	--	95.1	92.5	--	98.3	90.7
C.S. freeness, cc	723	678	670	395	375	380	633	617	567
% Change	--	-6.2	-7.3	--	-5.1	-3.8	--	-2.5	-10.4
Basis weight, lb/M ft²	13.6	13.3	13.4	13.4	13.5	12.8	13.2	13.2	13.5
Caliper, points	5.6	5.2	5.0	6.3	5.8	5.4	6.1	5.6	5.3
Apparent density	2.44	2.55	2.65	2.14	2.35	2.37	2.16	2.34	2.54
% Change	--	+4.5	+8.6	--	+9.8	+10.7	--	+8.3	+17.6
Bursting strength, psig	17.9	23.7	26.2	13.7	19.6	21.1	17.2	23.2	33.1
Factor	1.32	1.79	1.96	1.02	1.46	1.65	1.30	1.76	2.45
% Change	--	+35.6	+48.5	--	+43.1	+61.8	--	+35.4	+88.5
Mod. ring compression, lb/inch	3.8	4.1	4.5	4.3	4.8	4.6	3.8	4.0	4.8
Factor	0.278	0.309	0.336	0.322	0.358	0.359	0.284	0.307	0.353
% Change	--	+11.2	+20.9	--	+11.2	+11.5	--	+8.1	+24.3
Tear, grams	116.4	103.0	98.8	38.6	40.0	37.6	87.6	85.1	71.9
Factor	8.59	7.75	7.38	2.89	2.97	2.94	6.64	6.45	5.31
% Change	--	-9.8	-14.1	--	+2.8	+1.7	--	-2.9	-20.0
Tensile, lb/inch	12.2	14.8	16.2	12.2	15.2	15.1	12.4	15.6	20.0
Factor	0.90	1.12	1.21	0.91	1.13	1.18	0.94	1.19	1.48
% Change	--	+24.4	+34.4	--	+24.2	+29.7	--	+26.6	+57.4
Stretch, %	2.08	2.32	2.26	1.70	2.03	2.14	1.86	2.18	2.43
% Change	--	+11.5	+8.7	--	+19.4	+25.9	--	+17.2	+30.6
Et, lb/inch	1646	1899	2113	1675	1967	1989	1640	1943	2326
Factor	121.4	142.9	157.9	125.5	145.9	155.3	124.2	147.8	172.0
% Change	--	+17.7	+30.1	--	+16.3	+23.7	--	+19.0	+38.5
TEA, ft-lb/ft²	2.2	3.0	3.2	1.8	2.7	2.9	2.0	2.9	4.1
% Change	--	+36.4	+45.5	--	+50.0	+61.1	--	+45.0	+105.0
ZDT, psi	38.8	52.4	54.4	74.0	101.4	111.8	52.7	61.7	93.0
% Change	--	+35.1	+40.2	--	+37.0	+51.1	--	+17.1	+76.5
Brightness, %	14.4	17.9	23.6	22.2	26.6	28.8	16.6	20.2	33.4
% Change	--	+24.3	+63.9	--	+19.8	+20.7	--	+21.7	+101.2
Zero span factor, km	13.49	13.71	14.65	10.86	12.24	12.05	12.98	12.95	13.46
% Change	--	+1.6	+8.6	--	+12.7	+11.0	--	-0.2	+3.7

[a] Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

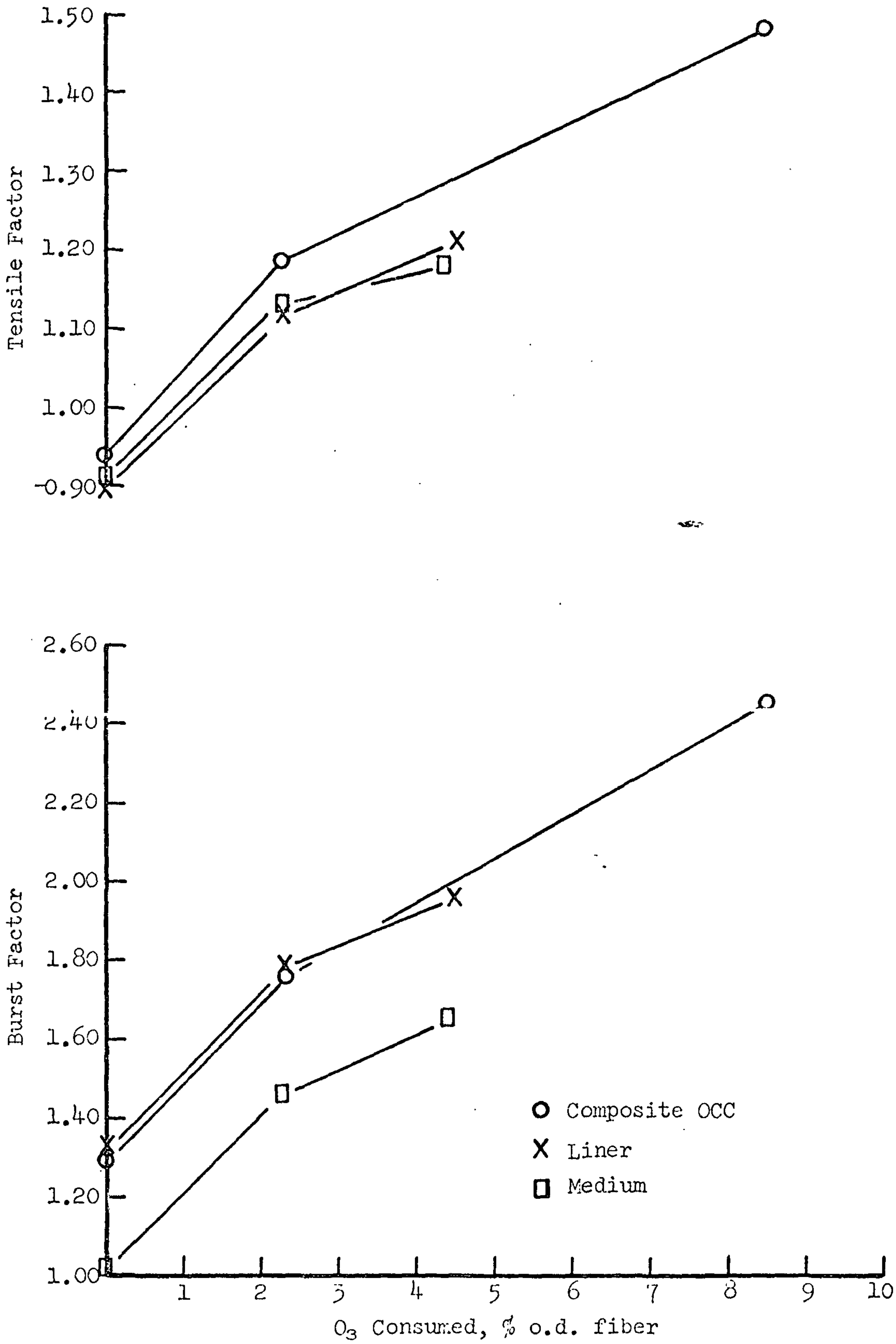

Figure 6. Burst and Tensile Factors on Ozonated Liner and Medium Fractions and Composite OCC

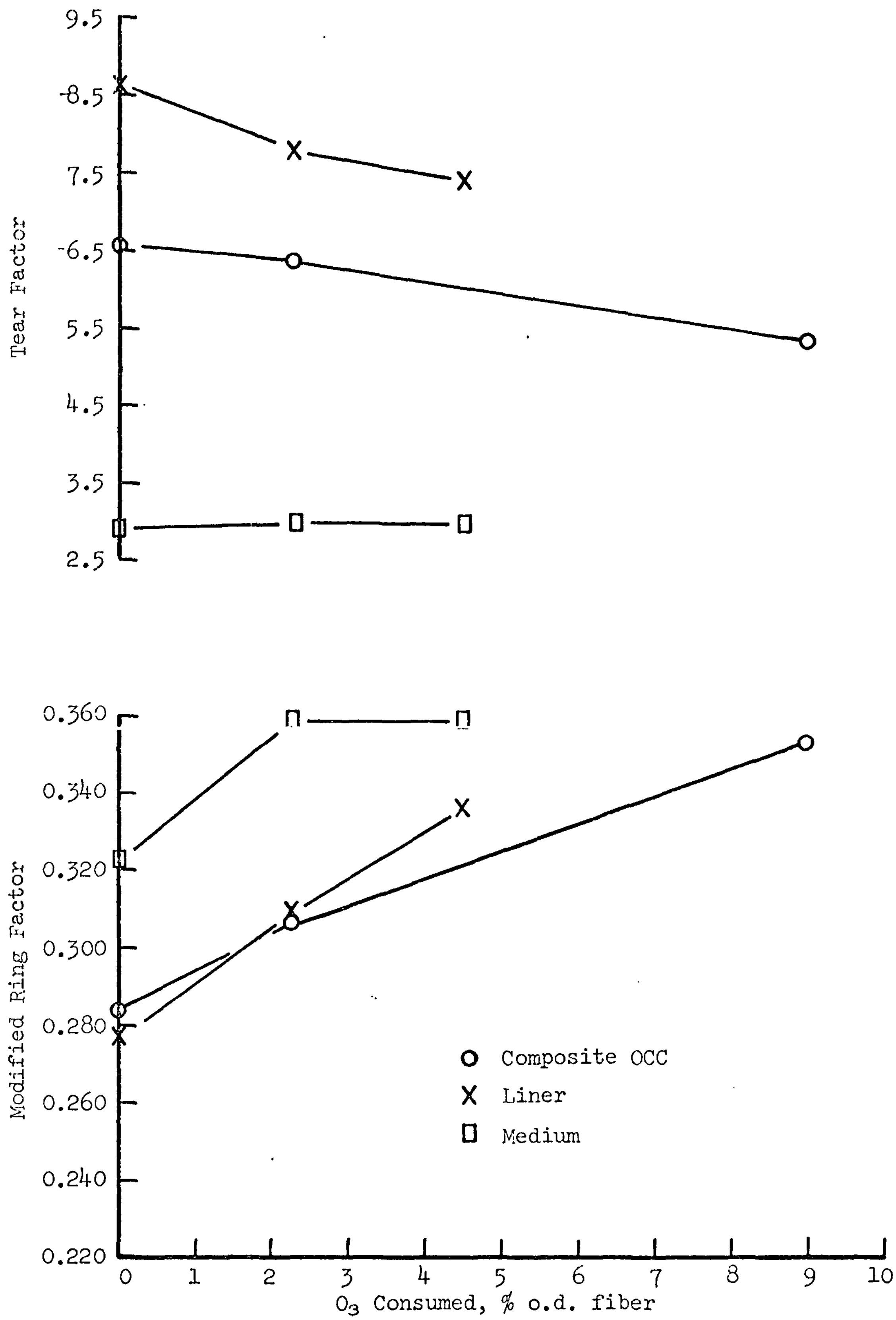

Figure 7. Tear and Modified Ring Results on Ozonated Liner and Medium Fractions and Composite OCC

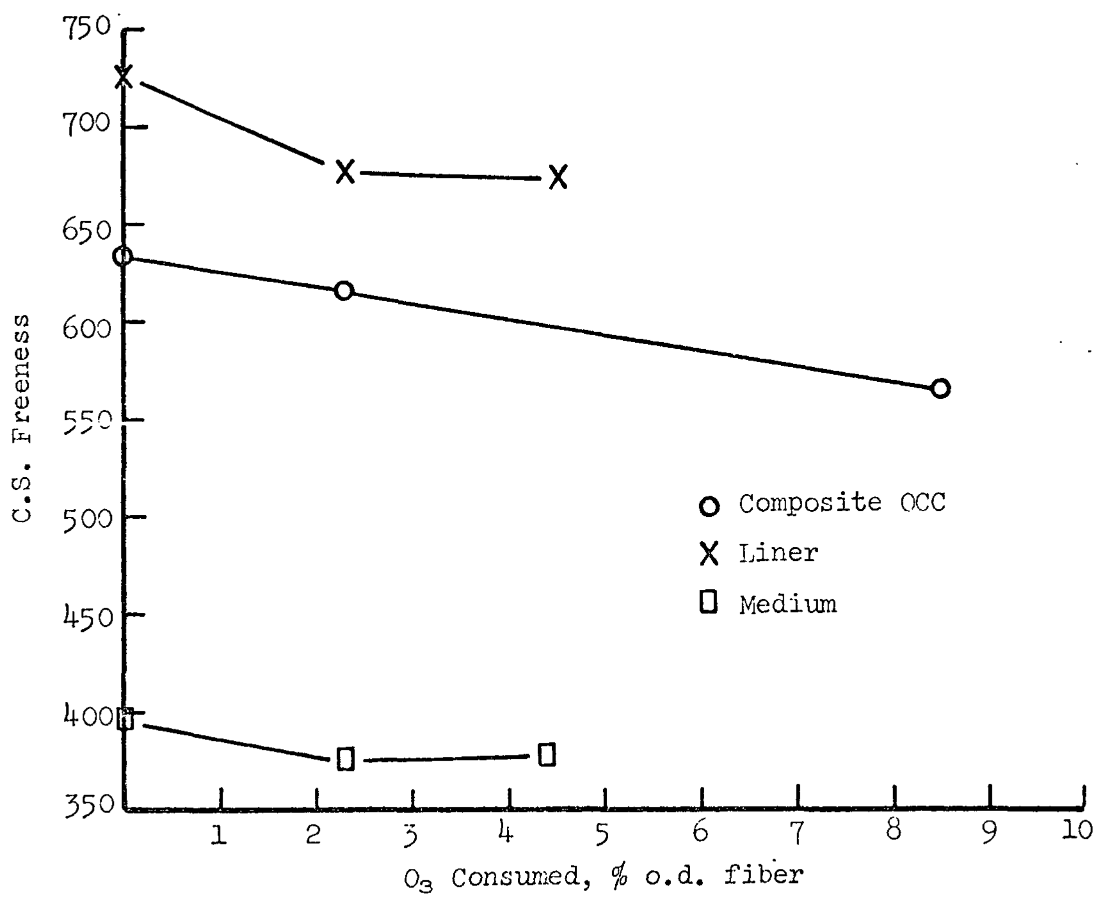

Figure 8. Freeness Results on Ozonated Liner and Medium Fractions and Composite OCC

both the liner and the medium show similar percentage physical strength increases suggests that fiber modifications are taking place which are not visually evident.

Blending of Ozonated Liner and Medium Fractions

Studies have been initiated to blend the untreated and ozonated liner and medium fractions in various combinations to determine the best ways to improve strength and minimize cost. Initial experimental trials have been completed but analysis of the data is incomplete at this time.

Reduction of Ozone Application Rate

Early ozonation studies were carried out utilizing approximately 2% ozone in oxygen at a flow rate of 4 liters per minute. Physical test results on handsheets prepared from the ozonated fibers receiving less than 2.5% ozone consumed tended to be erratic. It was speculated that the erratic results were due to the laboratory process used in ozonation of the fibers which failed to allow sufficient time for diffusion of the O_3/O_2 gas through the fiber mass. Reaction times of 5 to 10 minutes corresponding to approximately 0.8 and 1.5% O_3 consumed were potentially insufficient to achieve necessary equilibrium. To check this hypothesis, the process was modified to provide a flow rate of 1 liter per minute of oxygen containing 2% ozone. Effective reaction time intervals were thus increased by a factor of four for a given ozone consumption level.

Physical test results are shown in Table IV. The changes in strength at the low O_3 levels were about the same at the two O_3 addition rates. However, most of the strength properties increased with increasing O_3 as expected.

TABLE IV

COMPARISON OF PROPERTIES OF HANDSHEETS PREPARED FROM OCC OZONATED AT DIFFERENT O_3 RATES

O_2 Feed Rate (~2% O_3 in O_2)	1 Standard liter/min				4 Standard liters/min[a]			
Ozonation time, min	0	20	40	60	0	5	10	15
Ozone applied, % of o.d. fiber	--	0.732	1.539	2.261	--	0.820	1.61	2.35
Ozone consumed, % of o.d. fiber	--	0.731	1.535	2.252	--	0.814	1.59	2.31
Reaction efficiency, %	--	99.9	99.7	99.6	--	99.2	98.5	98.3
C.S. freeness, cc	660	615	630	610	633	647	627	617
% Change	--	-6.8	-4.6	-7.6	--	+2.2	-0.9	-2.5
Basis weight, lb/M ft^2	13.5	13.5	13.6	13.6	13.2	13.9	13.6	13.2
Caliper, points	6.0	5.7	5.7	5.7	6.1	6.4	6.1	5.6
Apparent density	2.26	2.37	2.39	2.41	2.16	2.18	2.21	2.34
% Change	--	+4.9	+5.8	+6.6	--	+0.9	+2.3	+8.3
Bursting strength, psig	16.6	2.37	2.39	2.41	2.16	2.18	2.21	2.34
Factor	1.23	1.41	1.50	1.64	1.30	1.36	1.49	1.76
% Change	--	+14.6	+22.0	+33.3	--	+4.6	+14.6	+35.4
Mod. ring compression, lb/inch	4.0	3.6	4.3	4.3	3.8	4.3	4.2	4.0
Factor	0.300	0.267	0.312	0.319	0.284	0.311	0.312	0.307
% Change	--	-11.0	+4.0	+6.3	--	+9.5	+9.9	+8.1
Tear, grams	86.4	92.4	94.0	89.6	87.6	56.7	98.5	85.1
Factor	6.39	6.85	6.90	6.57	6.64	6.93	6.87	6.45
% Change	--	+7.2	+8.0	+2.8	--	+4.4	+3.5	-2.9
Tensile, lb/inch	12.0	12.9	13.7	14.7	12.4	13.3	13.8	15.6
Factor	0.89	0.96	1.00	1.08	0.94	0.95	1.01	1.19
% Change	--	+7.9	+12.9	+21.4	--	+1.1	+7.4	+26.6
Stretch, %	2.04	2.07	2.04	2.20	1.86	1.96	2.11	2.18
% Change	--	+1.5	0.0	+7.8	--	+5.4	+13.4	+17.2
Et, lb/inch	1640	1735	1866	1922	1640	1767	1802	1946
Factor	121.3	128.6	137.0	141.0	124.2	127.0	132.5	147.8
% Change	--	+6.0	+12.9	+16.2	--	+2.3	+6.7	+19.0
TEA, ft-lb/ft^2	2.2	2.4	2.4	2.8	2.0	2.2	2.5	2.9
% Change	--	+9.1	+9.1	+27.3	--	+10.0	+25.0	+45.0
ZDT, psi	45.2	52.4	53.4	57.6	52.7	45.4	51.1	61.7
% Change	--	+15.9	+18.1	+27.4	--	-13.9	-3.0	+17.1
Brightness, %	16.4	17.1	19.0	20.2	16.6	18.2	19.1	20.2
% Change	--	+4.3	+15.9	+23.2	--	+9.6	+15.1	+21.7
Zero span factor, km	13.25	12.86	13.98	13.42	12.98	12.94	12.81	12.95
% Change	--	-2.9	+5.5	+1.3	--	-0.3	-1.3	-0.2

[a]Data obtained from Progress Report 1, Project 2697-53, entitled "Effect of Ozonation on Recycled Fiber", dated September 24, 1978

The C-stain examination of these treated fibers suggested there may be a more uniform treatment of the fibers at the lower oxygen flow rate, but the results were not completely clear-cut. SEM studies proved to be more interesting. It was noted that trends in fiber modification could be observed between the successive increments of ozone consumption. Visual changes on the fiber surface at the 0.7% level were very limited and could not be readily characterized. At the 1.5% level, a trend toward "pitting or erosion" of the surface was definite, but little or no surface fibrillation could be observed. At the 2.3% level, "pitting and erosion" increased and the first evidences of limited fibrillation were observed. It is speculated that fibers undergo a series of modifications during ozonation, namely,

1. An initial molecular surface reaction which cannot be physically observed,

2. Secondly, a pitting or erosion of the apparently smooth fiber surface occurs which causes the surface to become "spongy" and reduces rigid surface stresses, and,

3. Finally, the surface becomes sufficiently modified to reveal surface micorfibrils with reduced bonding to the parent fiber.

Since a general trend toward strength increase is shown even at the 0.7% ozone level, it may be suggested that all of the above changes affect one or more of the strength properties; but the rate at which a given property is increased may be related to a certain critical level of modification of the fiber surface. Early changes might be expected to modify fiber stiffness or conformability, but real increases in fiber surface may not be achieved until microfibrillation occurs. It appears that more fundamental studies are required to better understand ozone/fiber reactions. Current studies have suggested hypotheses for the reaction mechanism but, in reality, have raised additional questions that require answers before ozone/fiber reaction conditions can be fully defined and optimized.

Rapid Ozonation of Fiber Pads

Several screening trials were attempted to determine if ozonation would proceed rapidly in an excess of ozone. To achieve these goals, it was necessary to ozonate only small quantities of fiber in an appropriate reaction unit. Thin fiber pads of approximate 1-1/4 inch diameters were prepared in a Gelman filter unit at a consistency of 30-50%. Consistency was difficult to maintain but appeared to approximate consistencies normally achieved by fluffing. Pads contained about 35 mg of o.d. fiber. The pad was placed into an aluminum Gelman in-line filter which became the reaction vessel. Oxygen containing 2% ozone was applied to the pad at a flow rate of 4 liters per minute. Pads were treated for 1, 2, 4, and 8 minutes. Fibers were stained with C-stain. Color changes were compared with color changes occurring at known percentages of ozone consumed. Color changes on fibers from the 1 minute ozonation interval suggested the fibers consumed in excess of 2.3% ozone. At the 8 minute ozonation interval, color changes were roughly equivalent to 12% ozone consumed. Comparable time intervals under previous ozonation procedures were 15 to 90 minutes, respectively. The preliminary trials suggest that ozonation proceeds rapidly when sufficient ozone is present and processing conditions are optimized. Results indicate reaction rate was increased by a factor of ten. A continuous process based upon a moving web may be feasible.

Consistency Studies

The literature reports consistency of the pulp is critical to effective ozonation. Consistencies above 30% are reported to be necessary to provide reasonable reaction efficiencies. Therefore, a study has been initiated to determine the desirable consistency range for OCC pulp.

For this purpose, the OCC was centrifugally dewatered to about 30% consistency and then given a sufficient number of passes through the fluffer to obtain consistencies of about 30, 40, 53 and 67% prior to ozonation. The bulk densities of the so-treated pulps tended to vary inversely with the consistency, i.e., the higher the consistency, the lower the bulk density. Each pulp was ozonated using treatment times of 15 and 30 minutes corresponding to ozone consumptions of about 2.25+ and 4.0+%, respectively. It was observed during ozonation that a considerable degree of "balling" into fiber clumps occurred at the lowest consistency of 30%. The preliminary bursting strength results shown in Figure 9 indicate that the lowest results were obtained at the highest consistency at both O_3 treatment levels. This would be expected based on the literature. In general, it appeared that the highest results were obtained in the 40-50% consistency range which is also in accord with the literature. However, the 30% consistency results at the 2.25% O_3 level were not much lower than were obtained at the 40% consistency level. Perhaps, with a different reactor which minimized "balling", better results could be obtained in the 30-40% consistency range.

Effect of Fluffing

In previous and current work, the effects of ozonation on physical properties have been evaluated relative to the untreated "control" OCC

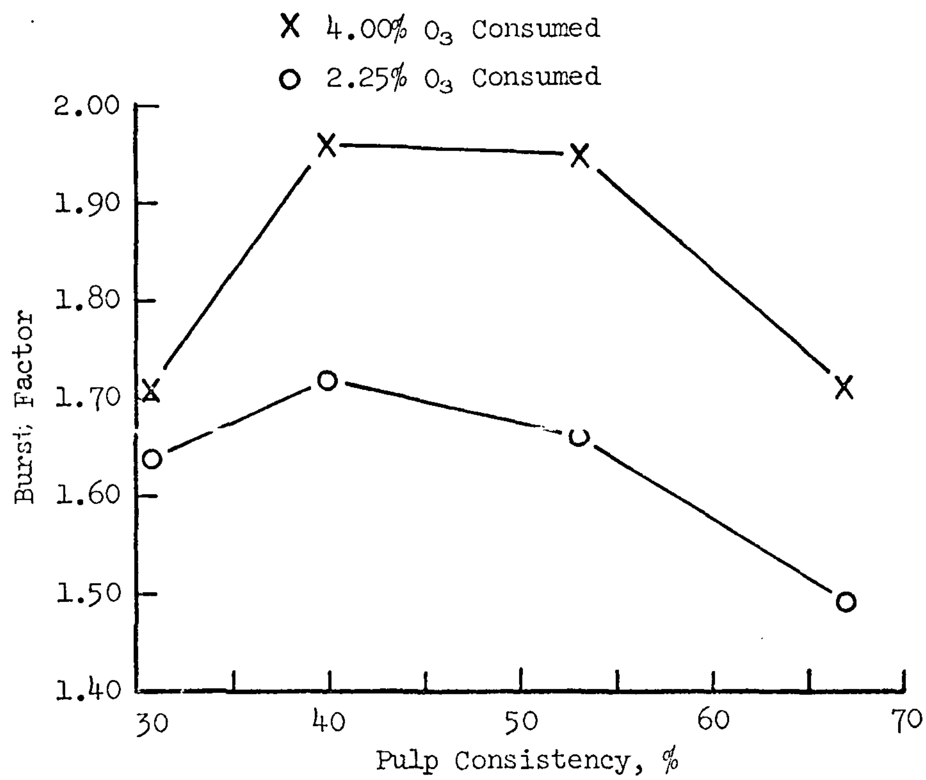

Figure 9. Effect of Ozonation Consistency on Burst Factor

pulp. The untreated pulp is processed and fluffed in the same manner as the ozonated pulps. There has been a question whether the laboratory fluffing lowers the physical properties of the "control" stock. If the "control" properties were lower than normal, this might result in exaggerated estimates of the effects of ozonation on physical properties.

A series of trials are in progress to evaluate whether the fluffing of the control stock has a degrading effect on physical properties. The results from these trials were not available at the time of this report. However, a preliminary comparison of results on fluffed and fluffed untreated OCC based on data at hand indicates that there is essentially no difference in physical properties due to the processing and fluffing of the pulp (see Table V). The fluffed OCC exhibited slightly higher burst, tensile, tear and stretch values, although none of the differences appear to be statistically significant. Thus, these data show no evidence that the laboratory fluffing of the untreated control had a degrading effect on physical properties.

There is a small difference in Canadian Standard freeness (30-35 cc) between the fluffed and unfluffed pulps. However, the freeness on these unrefined controls is sensitive to small variations in handling and dispersion and the difference obtained here may not be a significant factor.

<u>Patent Review</u>

A preliminary patent review is being prepared covering the ozonation process and equipment. It will also assist equipment design and planning for pilot scale operations should laboratory trials continue to be favorable.

TABLE V

EFFECT OF FLUFFING

	Unfluffed, Untreated OCC	Fluffed, Untreated OCC[a]	Difference, %[b]
C.S. freeness, cc	665	633	-4.8
Basis weight, lb/M ft^2	13.8	13.2	---
Caliper, points	6.1	6.1	---
Apparent density, lb/M ft^2/pt	2.25	2.16	-4.0
Bursting strength, psi	17.6	17.2	---
Factor	1.28	1.30	+1.6
Tensile, lb/inch	12.3	12.4	---
Factor	0.89	0.94	+5.6
Tear, grams	85.6	87.6	---
Factor	6.21	6.64	+6.9
Modified ring compression, lb/inch	4.2	3.8	---
Factor	0.304	0.284	-6.6
Stretch, %	1.73	1.86	+7.5
Et, lb/inch	1741	1640	---
Factor	126.3	124.2	-1.7

[a] Project 2697-53, Progress Report One, page 15.

[b] Based on unfluffed as reference.

Future Work

The current and future research on the ozonation process is divided into four parts, namely,

--Ozonation process variables

--Feasibility evaluation of low consistency treatments

--Optimization of ozonated and untreated fiber combinations

--Process alternatives and cost

The information developed in the first three parts is needed to properly assess the economic potentials of the ozonation process. As the process and furnish combination data is developed, the estimates of process and capital will be revised. Accordingly, the following research has been planned.

Current studies will be completed. They include:

1. Determination of the ozonation characteristics of commercial OCC from several sources.

2. The effect of blending ozonated liner and medium fractions on strength.

3. Determination of the effects of pulp consistency on the ozonation process.

In addition, studies covering the following are being planned:

1. Determination of the effects of pre- and post-refining on ozonated pulps.

2. Feasibility of low consistency treatments (may significantly lower capital costs).

3. Study of the strength properties of ozonated OCC/virgin pulp blends.

4. Effects of temperature variations on the ozonation process.

5. Determination of the nature and amount of possible soluble products resulting from ozonation.

CHEMICAL TREATMENTS AND ADDITIVES
(FKBG Funding)

A series of trials are underway to compare the effectiveness of various chemical treatments of recycled OCC with each other and with ozonation. The treatments under study and initial results obtained are briefly summarized below.

Caustic Treatments -- Atmospheric Pressure

Repulped OCC at 4% consistency was treated with NaOH using concentrations of 3 and 5% based on the weight of ovendry (o.d.) fiber. These concentrations were selected based on the work of Seifert and Long* who reported that an increase of about 25% in bursting strength resulted using 4% NaOH. Two temperatures were employed for this study, namely, room temperature (77°F) and about 198°F. The stocks were treated for two hours at each concentration and temperature level. Beating curves were obtained at each condition, as well as on the untreated control.

The yields for these caustic treatments ranged between about 94-96%. These yields are about the same as obtained by Seifert and Long.

Figure 10 shows that the caustic treatments at 198°F decreased the freeness by about 50 cc at each beating interval which is probably due to swelling of the fibers by the caustic. Figure 11 shows that the caustic treatments generally increased the burst and tensile factors at each beating interval. The percentage improvements in these properties tended to decrease as beating

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*Seifert, P. and Long, K. J., Tappi 57, no. 10: 69-72 (October 1974).

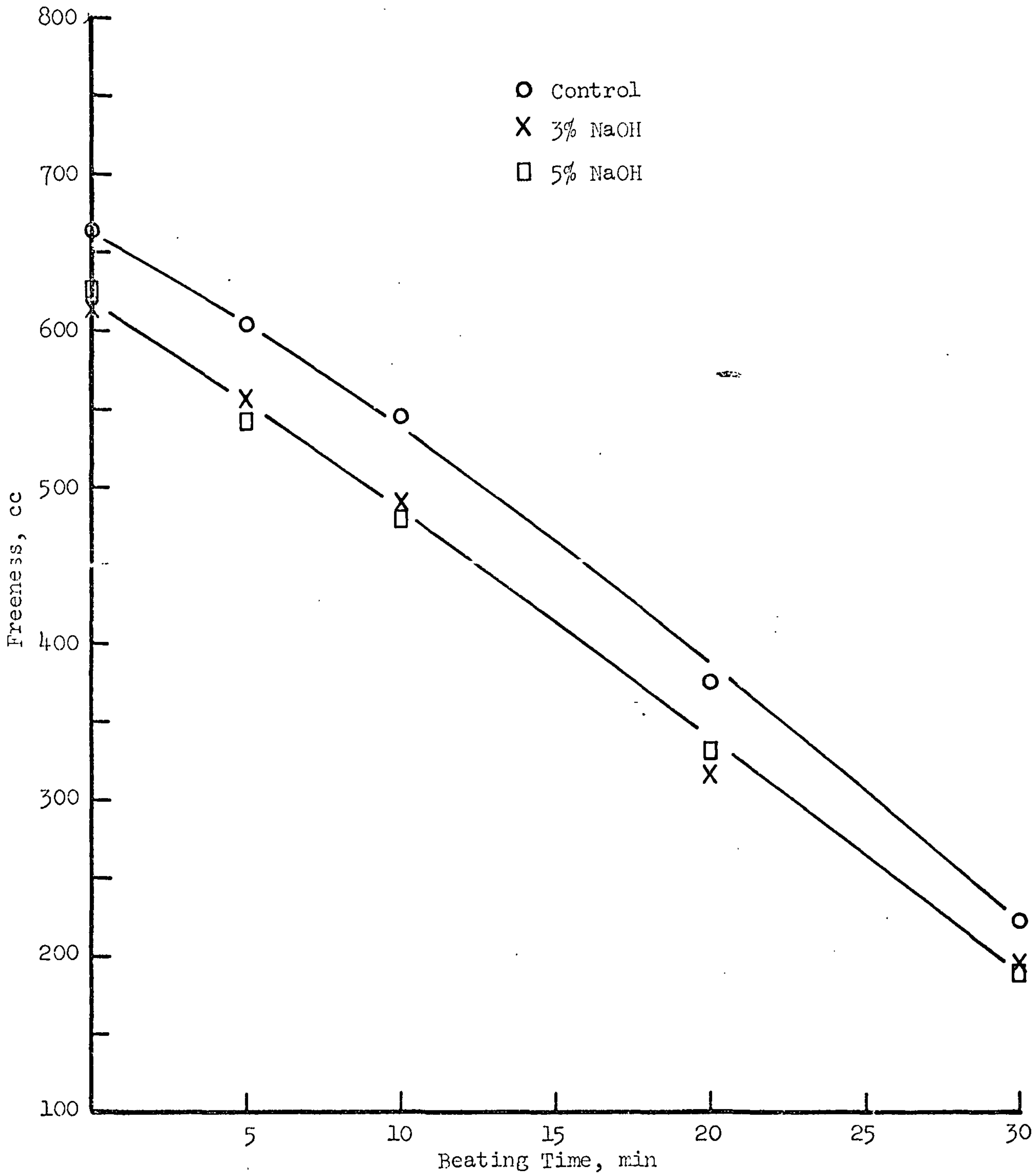

Figure 10. Effect of Caustic Treatments at Atmospheric Pressure on Freeness

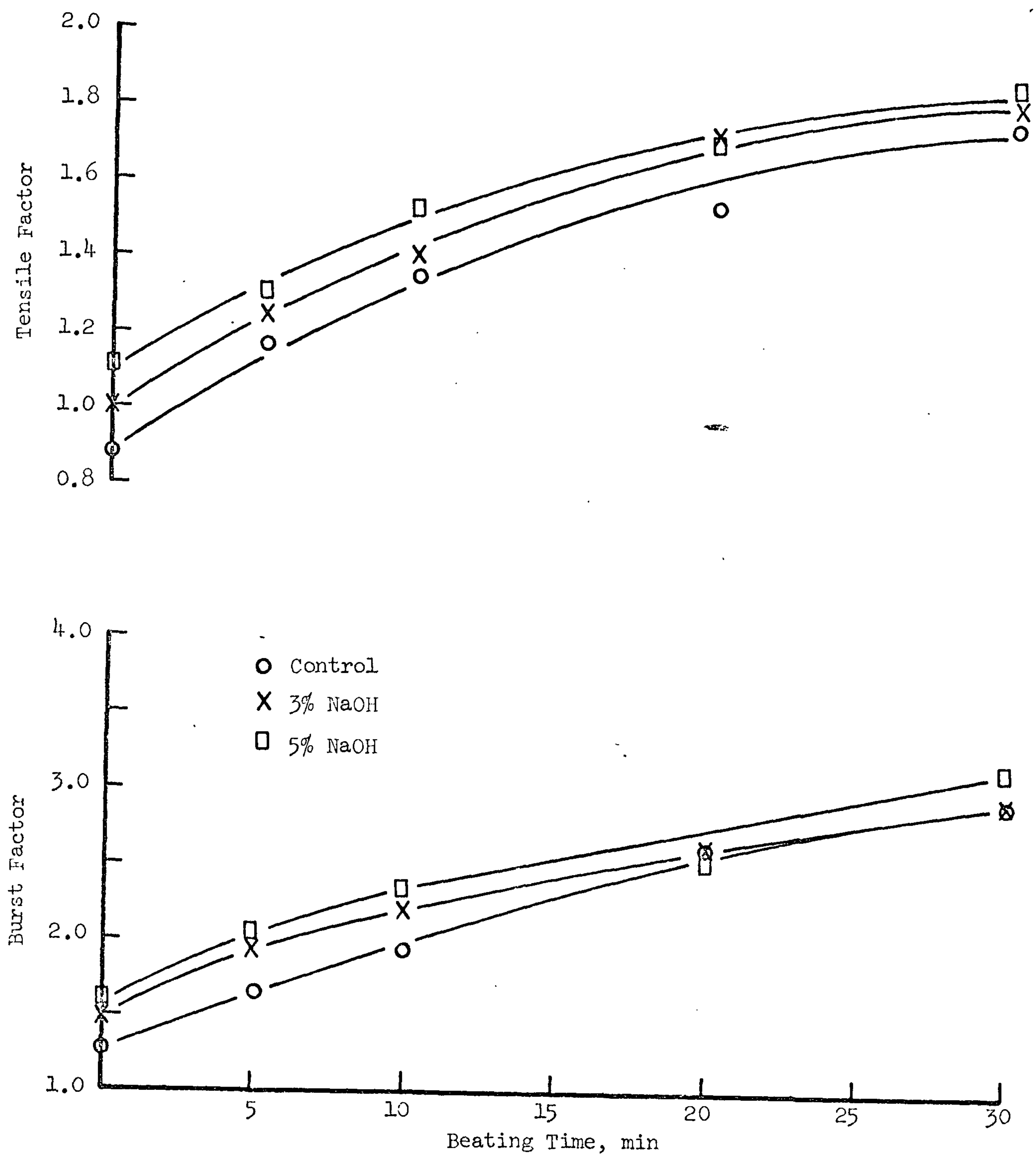

Figure 11. Effect of Caustic Treatments at Atmospheric Pressure on Burst and Tensile

progressed. It appears that the improvements in burst and tensile at the 0 beating time were approximately proportional to the caustic concentration (see Figure 12). Past work on ozonation indicated that a 35% improvement in burst could be achieved with 2.3% O_3 consumption. The results in Figure 12 suggest that caustic treatments near 7-8% might be required to achieve a 35% burst improvement. On the other hand, the tensile improvement of 25.8% with 5% caustic was approximately the same as that obtained at the 2.3% O_3 consumption level (27%).

The tear factor results in Figure 13 show that the caustic treatments tended to slightly increase the tearing strength at the shorter beating times. However, the differences are probably not significant. The effects of the caustic treatment on modified ring compression in Figure 13 are somewhat erratic which is probably due, in part, to the difficulties of carrying out compression tests on thin, light-weight sheets. With this reservation, the results suggest the caustic treatments tended to lower the modified ring strength in most cases at the various beating intervals.

When the burst and tensile factors are plotted against freeness, the results show that the control and caustic treated samples exhibit about the same test level at a given freeness level (Figure 14). This indicates that the increases in tensile and burst strength achieved with these caustic treatment conditions are about the same as would be obtained by mechanical refining.

The above trials were all carried out at low consistency. A limited trial is also in progress involving a high consistency caustic treatment of OCC. It has been speculated that such a treatment might have advantages

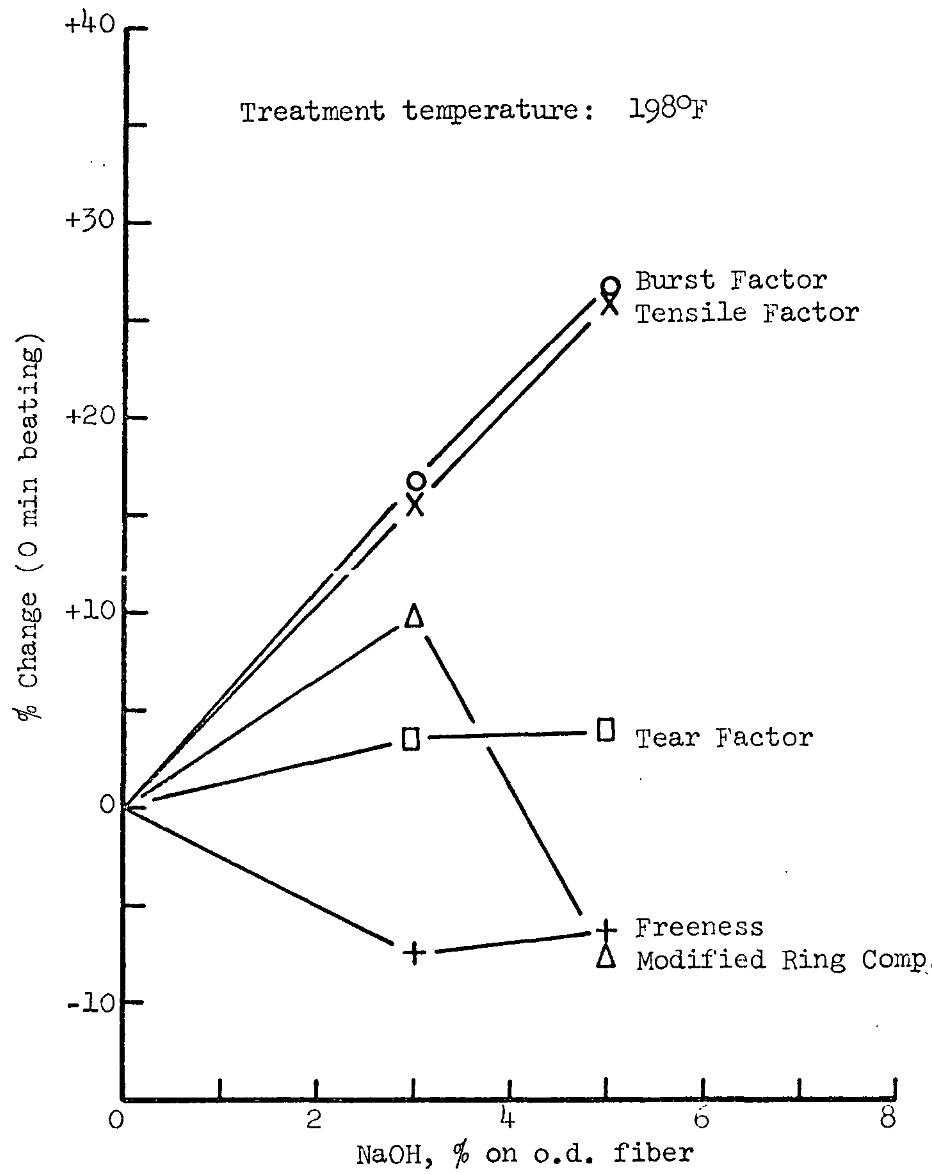

Figure 12. Effect of Caustic Treatment Concentration on Various Sheet Properties

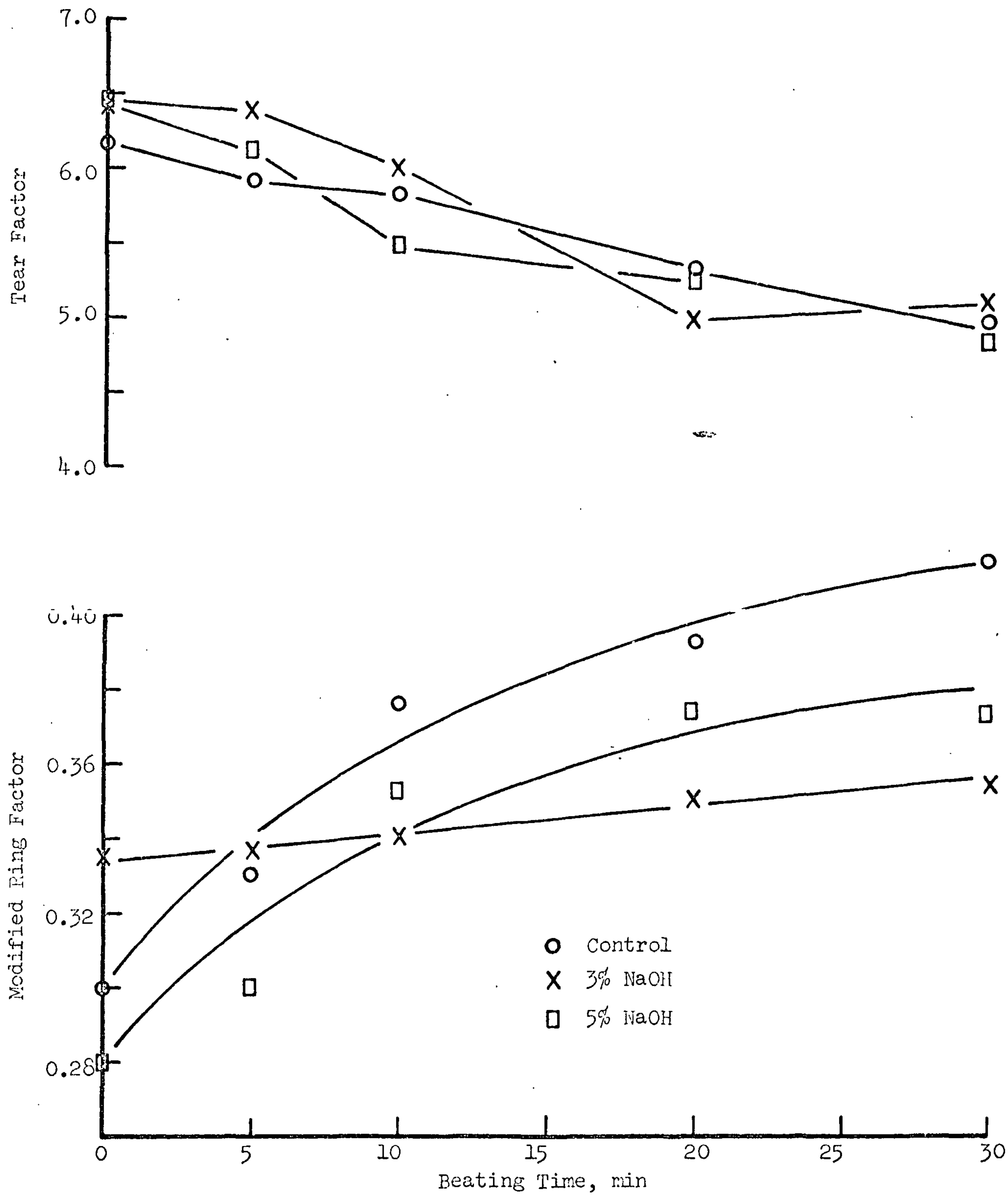

Figure 13. Effect of Caustic Treatments at Atmospheric Pressure on Tear and Modified Ring Compression

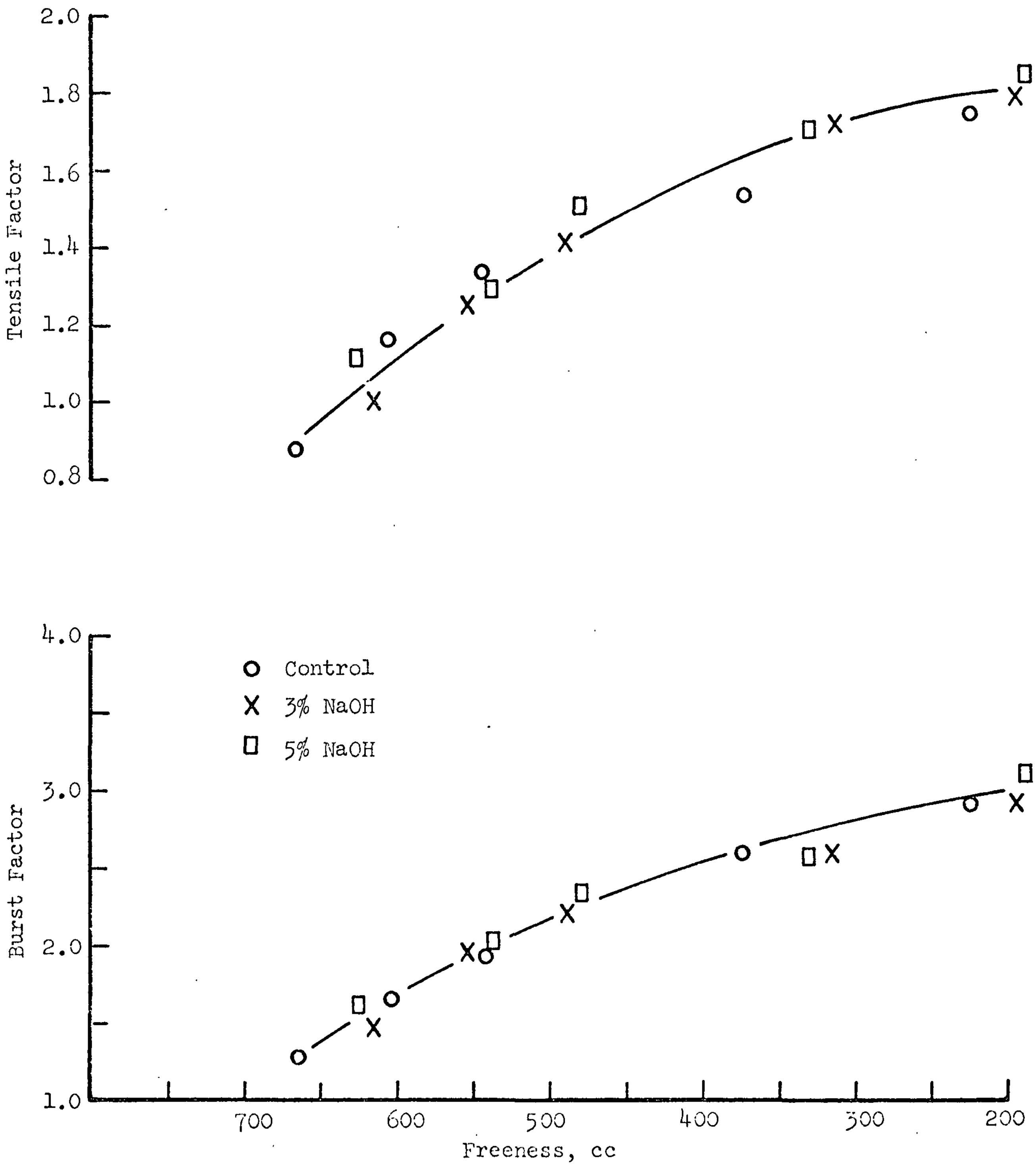

Figure 14. Tensile and Burst vs. Freeness on Caustic Treated OCC

for treating shredded OCC which is moistened to the desired consistency level. After treatment, the stock would be diluted for screening and cleaning.

Pressurized Chemical Treatments

The following treatments are being carried out in the Asplund defibrator to simulate the effect of chemical treatments in A/D systems. The processing has been carried out using 135 grams o.d. fiber with the selected amount of chemical. The consistency of the stock was about 25-30% with a dwell time in the Asplund preheater of 5 minutes with 100 psi steam. The following chemical levels have been used.

1. Caustic soda (NaOH): 3 and 5% on o.d. fiber
2. Sodium carbonate: 8 and 13.25%
3. Hydrogen peroxide (1%) in an alkaline slurry

In addition to the above, a mild oxygen treatment to give a moderate reduction in lignin content has been carried out. For this purpose, repulped OCC at about 32% consistency was treated with 90 psi O_2 at 110°C for 20 minutes. A small amount of magnesium ion was also added to prevent attacking the cellulose.

The above "cooks" have all been completed and the handsheet preparation at each beating interval is in process.

Chemical Additives

For comparison with the chemical treatments, including ozonation, a series of trials will be initiated using selected bonding and drainage aids to achieve acceptable strength and water-removal properties. These trials were deferred in order to carry out the chemical treatments; however, work on this phase will be started in early 1979. The initial materials are on hand and will include cationic corn starch, cationic potato starch, guar or locust beam gum and synthetic drainage aids.

At this time only a limited number of agents will be evaluated for the purpose of providing baseline comparisons with the various chemical treatments under study. In addition, trials may be carried out on ozonated or other chemically treated stocks in order to determine how well the agents would perform when applied to such stocks.

OCC and Virgin Primary at "Standard" Sheet Weight

In the ozonation and chemical treatment studies, the amounts of stock which could be conveniently processed were limited. Therefore, for strength comparison purposes, the handsheets were prepared using the standard handsheet weight (approximately 60 g/m^2 or 13 $lb/M\ ft^2$). Because portions of the initial work on this study were carried out using a heavier sheet weight (ca. 42 $lb/M\ ft^2$), it was desirable to obtain new beater curves on the OCC and virgin primary stocks to compare the effectiveness of the various treatments and additives. In addition, data were obtained at both 50 and 90 psi wet pressing levels at selected beater intervals in order to permit future comparisons of chemical treatments effectiveness with increased wet pressing.

The handsheet results on the OCC are shown in Tables VI and VII for the 50 and 90 psi wet pressing pressures, respectively. Tables VIII and IX contain the corresponding results on the virgin primary.

TABLE VI

OCC HANDSHEET PROPERTIES AT 50 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 0 min	5 min	10 min	20 min	30 min
C.S. freeness, cc	665	605	545	375	225
Basis weight, lb/1000 sq. ft	13.8	13.6	13.8	14.0	13.0
Thickness, mils	6.1	5.8	5.5	5.0	4.6
Apparent density, lb/1000 sq. ft-mil	2.25	2.36	2.52	2.78	2.79
Burst, psig	17.6	22.5	26.8	36.8	37.6
Factor	1.28	1.66	1.94	2.62	2.90
Modified ring, lb/inch	4.2	4.5	5.2	5.5	5.4
Factor	0.304	0.331	0.377	0.393	0.415
Tensile, lb/inch	12.3	16.1	18.6	21.6	22.8
Factor	0.89	1.18	1.34	1.54	1.76
Et, lb/inch	1741	2016	2101	2290	2374
Factor	126.3	148.2	152.1	163.5	182.8
Tear, grams	85.6	80.8	80.8	74.8	65.2
Factor	6.21	5.94	5.85	5.34	5.02
Stretch, %	1.73	2.09	2.14	2.51	2.56
TEA, ft-lb/sq. ft	1.8	2.8	3.5	4.6	4.9

TABLE VII

OCC HANDSHEET PROPERTIES AT 90 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 0 min	5 min	10 min	20 min	30 min
C.S. freeness, cc	665	605	545	375	225
Basis weight, lb/1000 sq. ft	13.8	13.9	13.8	13.7	13.8
Thickness, mils	5.9	5.4	5.1	4.8	4.6
Apparent density, lb/1000 sq. ft-mil	2.35	2.58	2.70	2.85	3.01
Burst, psig	18.0	24.5	29.4	35.2	39.9
Factor	1.30	1.76	2.13	2.57	2.90
Modified ring, lb/inch	4.0	4.6	4.8	5.4	5.5
Factor	0.290	0.331	0.348	0.394	0.399
Tensile, lb/inch	13.6	17.4	19.2	22.6	24.2
Factor	0.98	1.25	1.39	1.66	1.76
Et, lb/inch	1834	2113	2182	2318	2522
Factor	132.5	151.9	158.0	169.7	183.1
Tear, grams	87.6	82.0	77.6	67.6	60.4
Factor	6.33	5.89	5.62	4.95	4.39
Stretch, %	1.85	2.06	2.09	2.73	2.60
TEA, ft-lb/sq. ft	2.1	3.0	3.3	5.2	5.3

TABLE VIII

VIRGIN PRIMARY HANDSHEET PROPERTIES AT 50 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 5 min	20 min	35 min	50 min	60 min
C.S. freeness, cc	765	725	640	435	270
Basis weight, lb/1000 sq. ft	13.6	14.4	13.8	13.6	14.1
Thickness, mils	6.2	5.6	5.1	4.6	4.5
Apparent density, lb/1000 sq. ft-mil	2.20	2.55	2.72	2.93	3.12
Burst, psig	18.1	31.7	37.1	42.4	47.8
Factor	1.33	2.20	2.69	3.12	3.40
Modified ring, lb/inch	4.4	5.2	5.0	5.4	5.8
Factor	0.324	0.361	0.362	0.397	0.409
Tensile, lb/inch	13.3	19.3	21.7	25.1	28.6
Factor	0.98	1.34	1.58	1.85	2.03
Et, lb/inch	1868	2106	2246	2422	2765
Factor	137.2	146.5	163.0	178.1	196.7
Tear, grams	168.8	127.2	107.2	87.2	76.4
Factor	12.39	8.85	7.78	6.41	5.43
Stretch, %	1.43	2.00	2.48	2.73	2.47
TEA, ft-lb/sq. ft	1.5	3.1	4.6	5.8	5.7

TABLE IX

VIRGIN PRIMARY HANDSHEET PROPERTIES AT 90 PSI WET PRESSING PRESSURE

Physical Property	Beater Interval 20 min	35 min	50 min
C.S. freeness, cc	725	640	435
Basis weight, lb/1000 sq. ft	14.2	14.4	13.2
Thickness, mils	5.0	4.9	4.5
Apparent density, lb/1000 sq. ft-mil	2.80	2.97	2.97
Burst, psig	33.4	43.2	44.1
Factor	2.36	2.99	3.33
Modified ring, lb/inch	4.8	5.3	5.2
Factor	0.338	0.368	0.394
Tensile, lb/inch	21.5	24.6	25.9
Factor	1.52	1.70	1.96
Et, lb/inch	2264	2502	2756
Factor	159.9	173.2	208.2
Tear, grams	116.0	94.8	79.2
Factor	8.19	6.56	5.98
Stretch, %	2.23	2.42	2.73
TEA, ft-lb/sq. ft	4.0	5.0	5.3

Figures 15 and 16 compare the OCC and virgin kraft properties at the standard (50 psi) wet pressing pressure. As may be noted in Figure 14, the burst factor of the OCC is about 39% lower than the virgin kraft at an arbitrarily selected freeness level of 600 cc. This is about the degree of improvement obtained in past work with ozone treatments at the 2.3% O_3 consumption level. It is somewhat greater than was achieved with the 5% caustic treatment discussed previously.

Future Work

During 1979 it is planned to complete those parts of the research program involving addition of composite OCC to linerboard. This will include:

1. Chemical treatments -- Work in progress will be completed and additional agents evaluated as seems desirable.

2. Chemical additives -- The use of bonding and production aids either alone or in combination with refining will be evaluated.

3. Refining methods and techniques

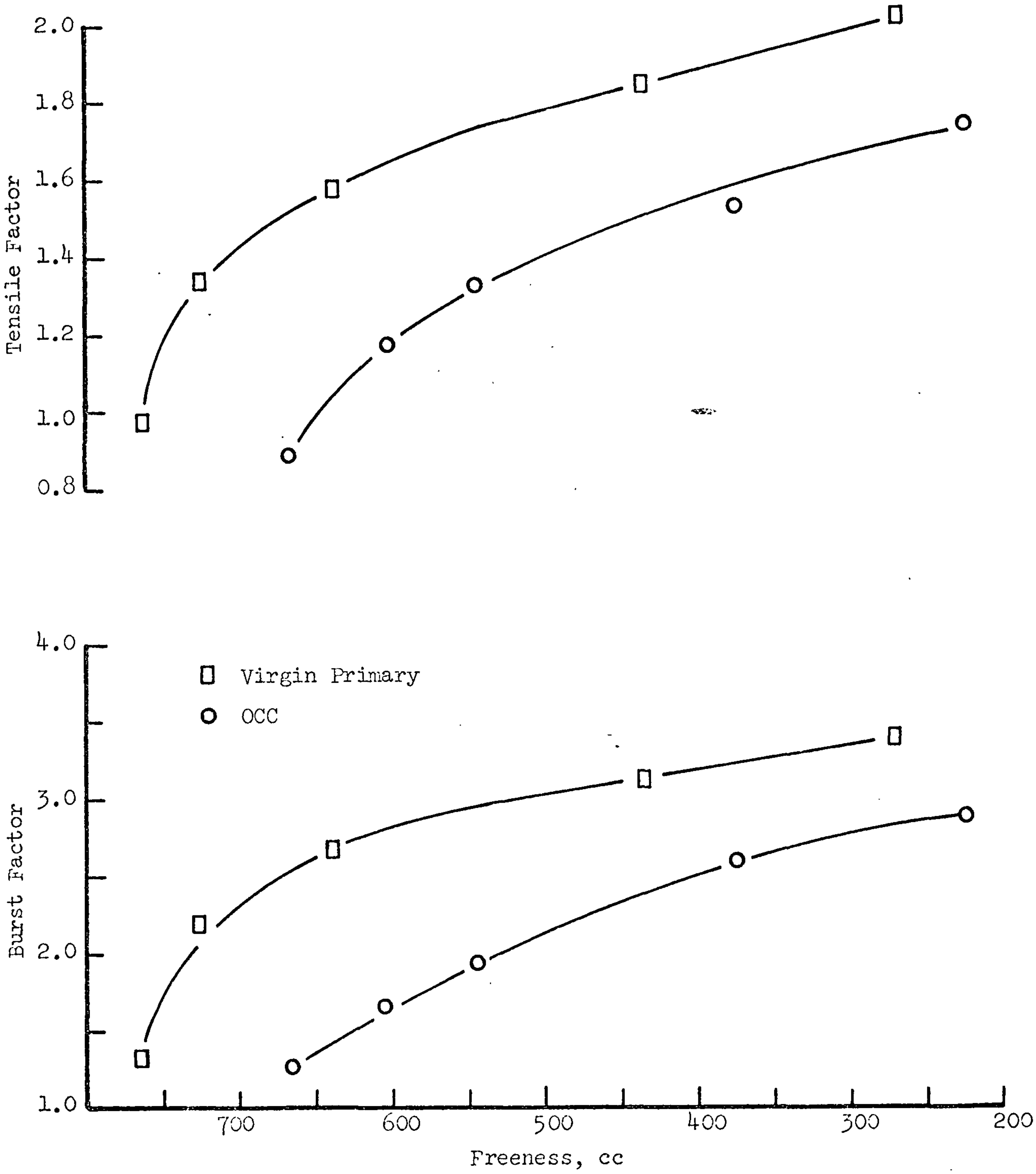

Figure 15. Tensile and Burst Properties on OCC and Virgin Kraft (50 psi wet pressing)

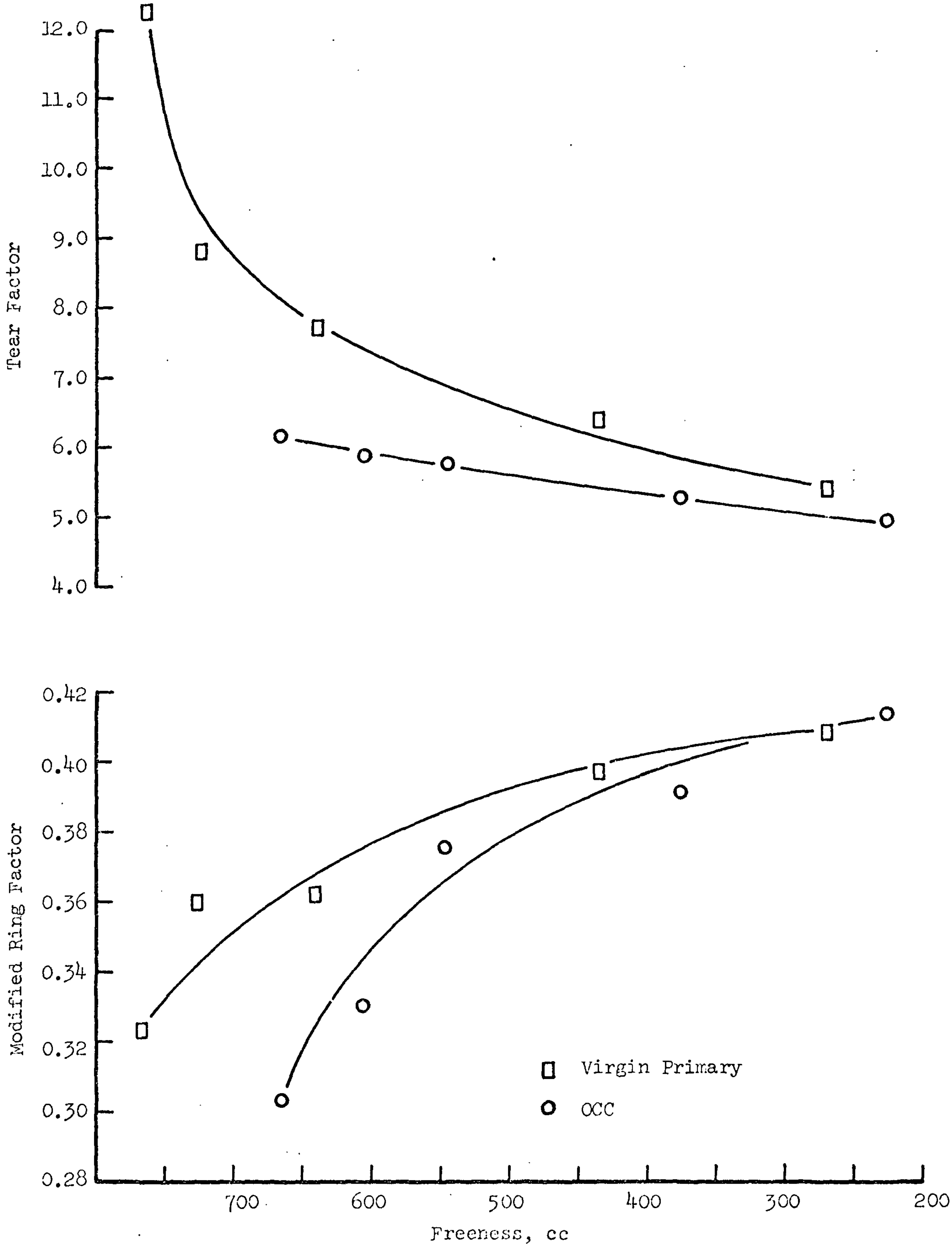

Figure 16. Tear and Modified Ring Compression Factors on OCC and Virgin Kraft (50 psi wet pressing)